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ORIGINAL ARTICLE

Synthesis and structural evolution of vanadium carbide in nano scale during mechanical alloying

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Abstract In this study, nano crystalline vanadium carbide was synthesized by mechanical alloying method. V_2O_5, C and Mg powders were placed in a planetary ball mill and sampled after different milling times. XRD and FESEM were used for characterization of synthesized powder. Studies showed that crystalline V_8C_7 has been synthesized by 24 h milling and subsequently heat treatment at 800 °C. It was concluded that the V_8C_7 crystallites were nano sized and the lattice parameter deviated slightly from the standard size. Furthermore, milling led to increase in strain and decrease of vanadium carbide particle size.

1. Introduction

The transition metal carbides have very high melting points, hardness and high temperature strength. These materials also exhibit good electrical and thermal conductivities. Among them vanadium carbide is the most attractive because of its many excellent physical and mechanical properties such as high hardness, excellent wear resistance, good corrosion resistance, excellent high temperature strength, high chemical and thermal stability even at high temperatures. It is commercially used in tool bits and cutting tools (Mahajan et al., 2013; Kurlov et al., 2013; Ye et al., 2009; Chen et al., 2011; Oelerich et al., 2001).

Presently, various methods for synthesizing vanadium carbide powders have been investigated including direct element reaction (Schwarzkopf and Kieffer, 1953), mechanical alloying (Dai et al., 2012), temperature programmed reaction (Lin et al., 2012), gas reduction-carburization (Mahajan et al., 2012; Zheng et al., 2012). Mechanical alloying (MA) as production process in cemented carbides has attracted many interests due to its capability of producing nano-crystalline powders prior to sintering (Dai et al., 2012; Zheng et al., 2012). This method has a number of potential advantages. MA process is simple, cheap and can be performed at ambient temperature. Mechanical alloying (MA) is a popular method to fabricate materials with

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novel structures and/or properties (Suryanarayana, 2001; Hoseinpur et al., 2013; El-Eskandarany, 2001).

Although some research were done for synthesizing vanadium carbide by mechanical alloying, but the effect of microwave heating after milling on the properties of synthesized materials was not investigated. So, this paper focused on synthesis and structure evolution during synthesis of V_8C_7 nano powder by MA method. Also the effect of microwave heating after milling on particle size, lattice parameter and phase formation of vanadium carbide was investigated.

2. Experimental

2.1. Materials and treatments

The starting materials were commercially available powders of V_2O_5 (purity of 99.9% and mean particle size of 200 μm), magnesium (purity of 97% and mean particle size of 100 μm) and amorphous graphite (purity of 99.8% and mean particle size of 50 μm). All the input materials with stoichiometric ratio were mixed according to the following reaction:

$$V_2O_5 + 5Mg + 2xC = 2VC_x + 5MgO \quad (1)$$

A SPEX ball mill with stainless vials (volume 250 ml) and balls (diameter 20 mm) was used for the mechanical milling. In order to protect the materials from oxidation, the vial was sealed with high-purity argon with a pressure of about 1 MPa. The ball to powder weight ratio was 20:1. Milling was carried out at a rotation speed of 250 rpm for 1, 3, 6, 12, 18 and 24 h.

Since vanadium carbide is chemically stable at room temperature and cannot be easily attacked even by strong acids, to remove the by-product (MgO), the as-milled powders were treated with 5% acetic acid solution. To complete phase formation, microwave heating was performed for 24 h milled samples.

The samples were placed into a microwave heater with power of 900 W and frequency of 2.45 GHz. A SiC crucible was used as a susceptor due to its efficient absorbance of microwave energy (Oghbaei and Mirzaee, 2010; Razavi et al., 2009).

2.2. Characterization

The synthesized powders were characterized by X-ray diffraction (Bruker D8) with the voltage and current of 40 kV and 30 mA, respectively, and Cu Kα radiation (λ = 1.54A°). The crystallite size was evaluated through the Williamson–Hall method (Eq. (2)) Razavi et al., 2009; Razavi et al., 2012 and the lattice parameter was also obtained using the Nelson–Riley method (Eq. (3)) Razavi et al., 2009; Razavi et al., 2012; Tsai et al., 1989.

$$b\cos\theta = \frac{0.9\lambda}{d} + 2\eta Sin\theta \quad (2)$$

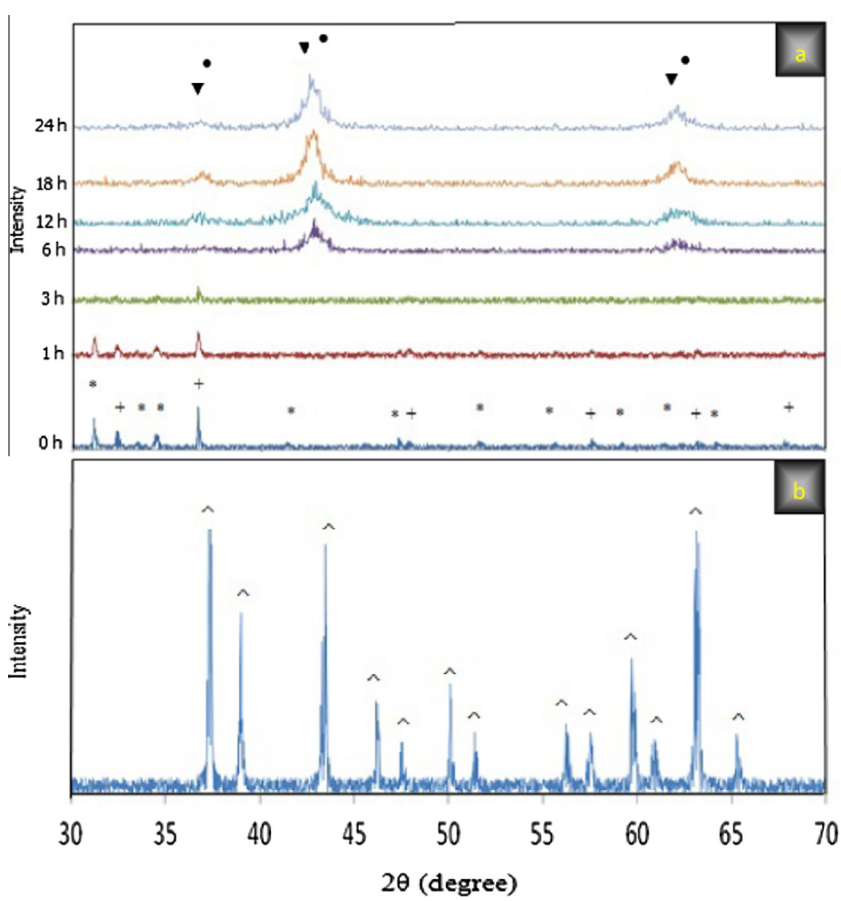

Figure 1 X-ray diffraction patterns of the (a) VC–MgO system which were milled in different times and (b) 24 h milled powders after microwave heating and leaching (V_4C_3 (●), MgO (▼), Mg (+), V_2O_5 (*), (V_8C_7 (ˆ)).